Transfer matrix method for precise determination of thicknesses in a 150-ply polyethylene composite material

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Abstract — **The multilayer structure of an ultra-high molecular weight polyethylene composite material was investigated in the terahertz (THz) spectral range by means of time domain spectroscopy (TDS) technique. Such structures consist of many alternating layers of fibres (~150), each being perpendicular to the other and each having a thickness of about 50 µm. A transfer matrix method (TMM) and a time-domain fitting procedure were used to determine thicknesses of all layers of the composite material with high accuracy.**

I. Introduction

Ultra-high molecular weight polyethylene (UHMWPE) consists of extremely long chains of polyethylene with a simple repeated atomic structure. Fibers with diameters of about 17 µm are extruded from the heated gel of UHMWPE using a spinneret and more than 95% of polymer chains have parallel orientation [1]. Next, flexible tapes made of a few about 50-70 µm perpendicular layers (often called plies) consisting of fibers are prepared. These tapes are stacked together and hot pressed to form a multilayer composite material with regular sequences of plies having mutually perpendicular orientations of fibers. Such composites are used in manufacturing bulletproof jackets, helmets and ballistic shields because of their high mechanical resistance. The determination of individual layer thickness as well as investigation of delaminations and other defects are crucial for reliable characterization of the manufacturing process and quality control. The THz range is well suited to study these composites, because UHMWPE has a low absorption coefficient varying between 0.3-1.5 cm^{-1} between 0.1 and 3 THz. Its refractive index of 1.53 is almost constant and thus the influence of dispersion is very small.

II. Characteristic of the Sample

The considered in this paper HB50 from Dyneema® tape consists of four alternately perpendicular plies of PE fibers (Fig. 1a) with diameters of about 17 µm each [1]. The thickness of a single ply is about 55-70 µm and a plastic rubber (17% of styrene-isoprene-styrene triblock copolymer (SISTC [1]) is used as a matrix. A long tape reel was cut into smaller sheets which were then layered and hot pressed to form a multilayer composite material. The photograph of HB50 composite samples having the dimensions of 50 x 50 mm^2 and thickness 9.45±0.1 mm is shown in Fig. 1b. The exact number of plies is not known to the authors.

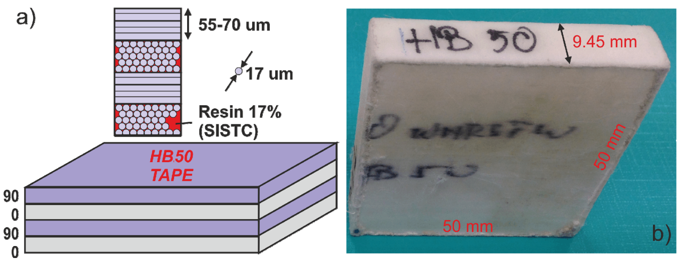

Fig. 1. The scheme of the HB50 tape (a) and the photograph of the manufactured composite.

First, we investigated the multilayer HB50 sample having smaller thickness of q=2.95 mm using a standard time domain spectroscopy system Spectra 3000 from TeraView. The complex refractive index N is equal to N=1.521+i·0.002. It was also proved [2] that a single layer of UHMWPE fibers exhibits a birefringence (Δn=0.04), which results in the dependence of the value of the refractive index on the orientation of the electric field vector in relation to the direction of fibers. This fact became the basis of the structure analysis in the experiment in the TDS reflection configuration.

III. Experimental Setup and Results

A THz TDS setup in reflection configuration with normal incidence was used for the investigation (Fig. 2a). The TDS setup uses a Ti:Sapphire laser, an InAs surface emitter, a GaAs photoconductive detector and a mechanical chopper. Figure 2b shows a THz pulse with FWHM=0.3 ps and side lobes. The 105-mm focal length resulting in a spot size at the surface of the sample of about 2 mm @ 1 THz.

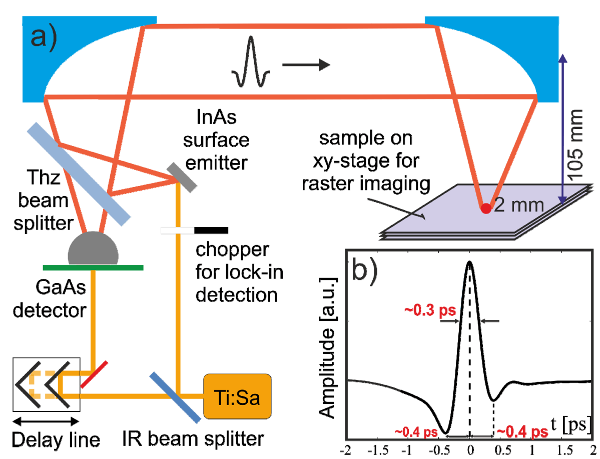

Fig. 2. The experimental setup (a) and the reference signal (b).

A bandwidth of the system is up to 3 THz with a dynamic range of about 40 dB. The setup is situated in a box purged with dry air, which provides low humidity (< 1%). The sample was placed on a rotating platform and the THz radiation was precisely focused on their front surfaces using a z-stage. The sample was also carefully aligned with respect to the parallel orientation of the electric field vector in relation to the direction of fibers of the front layer.

Figure 3 plots a typical waveform reflected from the sample with duration of about 140 ps with 13441 measuring points with spacing 0.0104 ps. In reflection geometry the propagating THz pulses are partly reflected from the front surface and from the interfaces between the media having different refractive indices (like layers/plies). Because of the multilayered structure of composite materials a sequence of pulses shifted in time are observed; each pulse coming from a reflection from a particular interface. In principle, knowing the refractive index of the single layer we can determine its thickness on the basis of the time difference between two pulses (their peaks) reflected from the front and back surfaces of this layer. However, for thin layers (~ 60 μm), pulses reflected from adjacent layers overlap in the time domain and cause errors in determining thicknesses of consecutive layers.

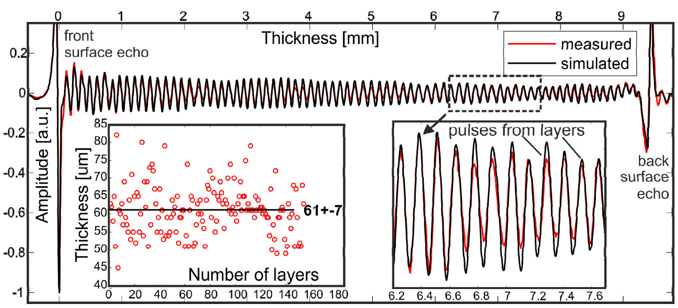

Fig. 3. Waveform reflected from the sample – measured and simulated. The inset on the left illustrates calculated thicknesses of particular layers.

IV. Simulation Results

The transfer matrix method and a time-domain fitting procedure [3] were used to the UHMWPE sample composed of about 150 layers with a quasi-binary profile of the refractive index, which means that values of the refractive index for subsequent layers are: $n+\Delta n_1$, $n-\Delta n_2$, $n+\Delta n_3$, …, respectively; $n \gg \Delta n_i$ and $\Delta n_1 \approx \Delta n_2 \approx \Delta n_3$… . The optimization of both parameters, i.e. the refractive index and the thickness for each layer independently would be computationally demanding. Therefore, we assume $\Delta n_1 = \Delta n_2 = \Delta n_3 = \Delta n$ and Δn was optimized for the whole sample, while calculations were limited to the optimization of thicknesses of layers. Due to the large number of layers, we significantly increased the optimization efficiency and accuracy by application of a hybrid fitting approach, which is based first on a brute force method for coarse searching followed by the accurate direct optimization.

Due to the complexity resulting from this partial superposition of pulses, the waveform reflected from the multilayer structure with the refractive index of i-th layer $N_i=n_i+i\kappa$; (i=1...k) can be simulated using the TMM [3]. The structure is surrounded from both sides by air with the complex refraction index $N_0=N_{k+1}=1$. In TMM each layer is modeled with the following two matrices:

- D_{ij} describing the behavior of the terahertz pulse at the interface between i-th and j-th ($j=i+1$) layer containing the corresponding Fresnel coefficients:

$$D_{i,j} = \frac{1}{t_{ij}}\begin{bmatrix} 1 & r_{ij} \\ r_{ij} & 1 \end{bmatrix}, \quad (1)$$

- P_i describing the propagation of the pulse through the i-th layer:

$$P_i(\omega) = \begin{bmatrix} \exp\left(\frac{i\omega N_i d_i}{c}\right) & 0 \\ 0 & \exp\left(-\frac{i\omega N_i d_i}{c}\right) \end{bmatrix}, \quad (2)$$

where ω is the angular frequency and c is the speed of light in vacuum. The multilayer structure can be described as the multiplication of all layers' matrices:

$$M_{total}(\omega) = \prod_{i=0}^{k} P_i(\omega) \cdot D_{i,i+1} = \begin{bmatrix} M_{11}(\omega) & M_{12}(\omega) \\ M_{21}(\omega) & M_{22}(\omega) \end{bmatrix}. \quad (3)$$

The transfer function for the reflection geometry ($R(\omega)$) can be calculated from the equation:

$$R(\omega) = \frac{M_{21}(\omega)}{M_{11}(\omega)}. \quad (4)$$

Finally, the simulated signal ($E_r(t)$) reflected from the multilayer sample can be calculated using the inverse numerical Fourier transform (F^{-1}) according to:

$$E_r(t) = F^{-1}\left[R(\omega) \cdot F\left[E_0(t)\right]\right], \quad (5)$$

where $F[E_0(t)]$ is the numerical Fourier transform of the incident THz pulse.

The time-domain fitting algorithm seeks for minima of:

$$QERR(d_1 \ldots d_k) = \sum_t \left[sign_{meas}(t) - sign_{sim}(d_1 \ldots d_k, t)\right] \quad (6)$$

QERR(d_1 ... d_k) describes the mean square deviation between the measurement and simulation. Thicknesses of individual layers are frequently varied until an optimal correlation between the measured and the simulated pulse is achieved.

Figure 3 (inset) shows optimal thicknesses calculated according to the proposed algorithm. We determined 154 plies with an average thickness of 61±7 μm. Almost 90% of calculated thickness values are within a range of 50-70 μm, what is in good agreement with other results. The waveform simulated basing on the calculated thicknesses agrees very well with the measurement with a correlation coefficient 0.97. As a result, we were able to determine the thicknesses of all layers of the multilayer (154 plies) structure by means of the reflection TDS technology with high accuracy.

References

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